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Committee of Experts on the Transport of Dangerous Goods and on the Globally Harmonized System of Classification and Labelling of Chemicals

Sub-Committee of Experts on the Transport of Dangerous Goods

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Geneva, 23 June – 2 July 2014
Item 2 (c) of the provisional agenda
Explosives and related matters:
review of tests in parts I and II of the Manual of Tests and Criteria

Manual of Tests and Criteria

Review of Test Series 8

Transmitted by the Australian Explosives Industry and Safety Group Inc. (AEISG)[1]

Introduction

1. At the forty-first session of the Sub-Committee (TDG), 25 June – 4 July 2012, the Chairman of the Working group on Explosives submitted a paper on the "Difficulties in carrying out classification tests" (refer INF.26, 2012), outlining:

- There was agreement that the problem of specifications in the test procedures was real and should be corrected;
- There could be other problems such as errors in procedure, incorrect use of the examples in the procedures, and a difficulty in identifying the key parameters of the tests;
- The Sub-Committee had already agreed that it should conduct a review of the tests mentioned in Parts I and II of the Manual with a view to:

[1] In accordance with the programme of work of the Sub-Committee for 2013-2014 approved by the Committee at its sixth session (refer to ST/SG/AC.10/C.3/84, para. 86 and ST/SG/AC.10/40, para. 14).

(a) Better defining the specifications of the tests;

(b) Better defining the tolerances associated with those specifications; and

(c) Removing any unnecessary or over-specifications;

2. This position was endorsed by the Sub-Committee of Experts on the Globally Harmonized System of Classification and Labelling of Chemicals (GHS) (see ST/SG/AC.10/C.4/42; paragraph 9).

3. The Working Group on Explosives discussed the issues raised in INF.26 and reported back to the TDG Sub-Committee (refer INF.67, 2012) recommending:

– There was general support for the way forward proposed by the chairman of the working group. The working group noted that IME (with the United States of America and Canada) is already underway in regards to reviewing Test Series 6 and that AEISG had committed to work on Test Series 8. The expert from the United Kingdom commented that they may also have resources that could be devoted to the project. The working group agreed with the priorities put forward by the chairman. The chairman of the working group reconfirmed his willingness to coordinate this effort.

4. The working group endorsed the proposal of informal document INF.26 that the sub-committees TDG and GHS accept the general principle outlined in regards to Parts I and II of the Manual, and include this activity in their next programme of work, and take action as deemed appropriate.

5. The TDG Sub-Committee considered the report (INF.67) and accepted the general principle outlined as regards Parts I and II of the Manual of Tests and Criteria and agreed to include this activity in its next programme of work, subject to concurrence by the GHS Sub-Committee (ST/SG/AC.10/C.3/82, para. 39) .

Discussion

6. In line with the principles outlined above, the various tests of Test Series 8, in Section 18 of the Manual of Tests and Criteria, have been reviewed with an amended version proposed in the attached Appendix for consideration by the Sub-Committee as part of their program of work.

7. It is not suggested or proposed that any changes recommended for the individual tests in Section 18 of the Manual of Tests and Criteria should be reflected or incorporated into any other similar tests in other sections of the Manual. The changes proposed for the tests in Section 18 apply solely to the testing of ammonium nitrate emulsions or suspensions or gels which are specifically subject to this section.

8. Section 18: Test Series 8

(a) 18.2 Test Methods

– An additional note has been added to Table 18.1 indicating that the Koenen Test, 8(c) is not required if Test 8(d) has been carried out with a negative (-) result. As both test types test the sensitiveness of the substance to the effects of heating under confinement, it is considered an unnecessary duplication of testing if both are conducted. It is not uncommon for ANEs to be transported in bulk (portable tanks) and hence Test 8(d) may be conducted as a necessity.

(b) 18.3 Test conditions

– "Unless otherwise specified" has been added as the tests do indeed specify test conditions which are not "the highest transport temperature". The term "highest transport temperature" has been amended for clarity and to be consistent with the phrase used in the tests (refer 18.4.1.1.1 and 18.4.1.1.2).

(c) 18.4 Series 8 Type (a) test prescription

(i) 18.4.1.1.1 and 18.4.1.1.2 are considered contradictory and incorrect as to the proper purpose of this test. Hence they have been combined to indicate that the test is one of thermal stability.

(ii) The term "Dewar vessel" has been removed throughout, replaced by "insulated test vessel" and the Figure 18.4.1.1, and any reference to it, has been removed. It is considered by various testing agencies that any insulated test vessel with the appropriate heat loss characteristics specified would be satisfactory.

(iii) A new 18.4.1.1.2 has been added to indicate that this test is not considered necessary where the ANE is manufactured at temperatures which are 20°C or higher than those experienced during transport.

(iv) In 18.4.1.2 the various terms "Test chamber", "Test cell", "oven", have been made consistent. The safety specifications have been replaced by a more general safety reference relevant to this test, with an indication that a number of tests may be run concurrently.

(v) The specification of +/- 1°C for the test chamber has been replaced by +/- 2 °C. This was considered an over-specification and is consistent with Test 3(c) where an even smaller increase in sample temperature is being sought.

(vi) The term "approximately" has been added to specifications which are not considered to require such precision.

(vii) References to "height" of the sample in the test vessel, "mass" of the sample, "time taken", "percentage mass loss", "change in composition", have all been removed as irrelevant for ANEs. 18.4.1.2.7 has been removed completely for the same reasons.

(viii) For clarity, "within the seven day period" has been added to the test criteria for a 6°C rise in sample temperature.

(d) 18.5 Series 8 Type (b) Test prescription

(i) For clarity, references are made to the explosives booster charge as the "donor charge" and the sample substance as the "acceptor charge".

(ii) The reference to "United Nations Standard detonator or equivalent" has been maintained only as an example (what is it, and why?). In the Appendix to the UN Manual of Test and Criteria, the examples have different base charges and are electric (Why?). The detonator used should be of sufficient strength to effectively initiate the booster.

(iii) The specification for the donor charge has been amended to allow for alternate, but at least equivalent, compositions which may be more readily available.

(iv) The "seamless" specification for the steel tubing has been removed as this is considered an over-specification for what is a sensitivity test (ie fragmentation of the tubing is not an issue here).

(v) In 18.5.1.2.1(d) reference to the diameter of the sample is removed.

(vi) In 18.5.1.2.1(e) references to the shock pressures, Table 18.5.1.1 and Figure 18.5.1.2 are removed as irrelevant here. The particular Table and Figure referenced are also removed as irrelevant here (these may be considered as useful information for the Appendices).

(vii) The term "approximately" has been included where specifications are considered less important.

(viii) Wood blocks, or similar, are added to the apparatus to ensure the whole assembly is mounted at least 100 mm off the ground.

(ix) Guidance has been added in 18.5.1.3.1 to sealing the bottom end of the tube to accommodate the sample and to ensure the assembly is held firmly in alignment.

(x) The reference to conducting the test over water has been removed but replaced with a method to ensure the witness plate is raised at least 100 mm off the ground, with clear space under the plate where the assembly sits.

(xi) The reference to the gap length in 18.5.1.4 has been removed as unnecessary.

(xii) Figure 18.5.1.1 has been updated to reflect the test set up.

(e) 18.6 Series 8 Type (c) Test prescription

(i) In 18.6.1.2.1 reference to various diameter orifice plates is removed with only those relevant to this test retained i.e. 1.5 mm for the calibration and 2 mm for the test.

(ii) In 18.6.1.2.1(a) the need for tubes in a test sequence to be of similar weight has been removed as this is irrelevant here.

(iii) Alternative gas fuels have been included, with propane as an example, provided the relevant heating rate can be assured.

(iv) A new 18.6.1.2.4 has been added recommending the tests be recorded.

(v) The lubricant has been generalised, with molybdenum disulphide kept as an example.

(vi) In 18.6.1.3.3 the time to reaction has been removed as irrelevant here. Reasoning has been added for collecting and weighing of tube fragments.

(vii) In 18.6.1.3.5 reference to various diameter orifices has been removed. Guidance has been added, following extensive testing, on the potential of solids blocking the orifice leading to false positive results with some ANEs. An allowance to repeat the test where this is suspected has been included (videoing of tests would assist here)..

(viii) Given the previous comment, the test criteria in 18.6.1.4 has been amended to expect at least three negative results out of a maximum of five tests.

(ix) Figures have been updated to provide more guidance, clarity.

(f) 18.7 Series 8 Type (d) Test prescriptions

Test 8(d)(i)

(i) The purpose of the test as outlined in 18.7.1.1 has been expanded for clarity of meaning.

(ii) In 18.7.1.2(a) a quality standard has been added for the welding of the test assembly. The specification for the steel has been included here – taken directly from the Figure 18.7.1.1.

(iii) The term "approximately" has been included where considered relevant for less precise specifications.

(iv) As suggested within the Working Group on Explosives discussions at the forty-third session, details for the fire have been made less prescriptive, however more evidentiary provisions have been added to ensure sufficient intensity of the fire is achieved, including:

- Attainment of a temperature of 800°C measured at the base of the pipe;
- Use of thermocouples, positioned externally at the pipe base, to record temperature profile of the fire (variation of fire temperature below 800°C during the test is considered normal and should not invalidate the test);
- Fuel to extend beyond the pipe in every direction (without specifying a distance) to ensure the fire fully engulfs the pipe;
- Burning time to be at least 30 minutes, with evidence of reaction included in 18.7.1.2(c);
- The observations made during the test have been extended to ensure validity of the test;
- Test to be recorded in colour for additional evidence. As a consequence, the specific fire type examples provided in 18.7.1.3.2, 18.7.1.3.3 and 18.7.1.3.4 have been removed.

(v) Means of measuring wind speed have been included (e.g. anemometer).

(vi) A new 18.7.1.4 has been added outlining safety issues relevant to the conduct of this test. It should be determined as a principle whether such additions are appropriate within the Manual.

(vii) The test criteria, now in 18.7.1.5, have been expanded, with the intention of providing clarity and to avoid misinterpretation of results.

Test 8(d)(ii)

(i) The purpose of the test as outlined in 18.7.2.1 has been expanded for clarity of meaning.

(ii) In 18.7.2.2(a) a quality standard has been added for the welding of the test assembly.

(iii) Flexibility has been afforded to specifications not considered to be of Significance e.g. metal stand, wind shield, fuel gas type, vent sizes.

(iv) Means of measuring wind speed have been included (e.g. anemometer).

(v) A new 18.7.2.5 has been added outlining safety issues relevant to the conduct of this test. It should be determined as a principle whether such additions are appropriate within the Manual.

(vi) The need to perform the test twice as outlined in 18.7.2.4.8 has been removed to maintain consistency with Test 8(d)(i).

Proposal

9. AEISG presents an amended version of Section 18 of the Manual of Tests and Criteria, covering Test Series 8 tests for ammonium nitrate emulsions or suspensions or gels, for discussion and proposed adoption.

10. The proposed modification of Section 18 of the Manual of Tests and Criteria is outlined in the attached Appendix and has been prepared using Section 18 of the Manual of Tests and Criteria, Fifth revised edition, with incorporated amendments to Section 18 already adopted and included in Amendment 2 to the Manual published in 2013.

Appendix

Section 18

Test Series 8

18.1 Introduction

The assessment whether a candidate for "ammonium nitrate emulsion or suspension or gel, intermediate for blasting explosives (ANE)" is insensitive enough for inclusion in Division 5.1 is answered by series 8 tests and any such candidate for inclusion in Division 5.1 should pass each of the three types of tests comprising the series. The three test types are:

Type 8 (a): a test to determine the thermal stability;

Type 8 (b): a shock test to determine sensitivity to intense shock;

Type 8 (c) or 8(d): a test to determine the effect of heating under confinement;

Test series 8 (d) has been included in this section as one method to evaluate the suitability of ANEs for the transport in portable tanks.

Where Test 8(d) is conducted, the results may be used in lieu of Test 8(c) as part of the assessment for inclusion in Division 5.1. Test 8(c) would therefore not be required.

18.2 Test methods

The test methods currently used are listed in Table 18.1.

Table 18.1: Test methods for Test Series 8

Test code	*Name of Test*	*Section*
8 (a)	Thermal Stability Test for ANE[a]	18.4
8 (b)	ANE Gap Test[a]	18.5
8 (c)	Koenen test[a,c]	18.6
8 (d)	Vented pipe tests[b]	18.7

a) This test is intended for classification.

b) These tests are intended for evaluating the suitability for transport in tanks.

~~b)~~c) This test is not required where the 8(d) test is conducted and a negative (-) result is achieved.

18.3 Test conditions

18.3.1 Unless otherwise specified in these tests, ~~T~~the substance should be tested as offered for transport, at the maximum temperature which may occur during transport~~highest transport temperature~~ (see 1.5.4 of this Manual).

18.4 Series 8 Type (a) test prescription

18.4.1 Test 8 (a): Thermal stability test for ammonium nitrate emulsions, suspensions or gels

18.4.1.1 Introduction

~~18.4.1.1.1~~ 18.4.1.1.1 This test is used to ~~measure the stability of~~determine whether a candidate for "ammonium nitrate emulsion, suspension or gel, intermediate for blasting explosives" ~~when subjected to elevated thermal conditions to determine if the emulsion is too dangerous to transport~~.

~~18.4.1.1.2 This test is used to determine whether the emulsion, suspension or gel is~~ thermally stable at temperatures encountered during transport. In the way this type of test is normally carried out (see 28.4.4), the 5000~~.5 litre~~ml ~~Dewar~~insulated test vessel is only representative for packagings, IBC's and small tanks. For the transport of ammonium nitrate emulsions, suspensions or gels the test ~~can be~~is used to measure ~~its~~their thermal stability during tank transport if the test is carried out on candidate products which are at a temperature 20 °C higher than the maximum temperature which may occur during transport, ~~including~~or if higher, at the temperature at the time of loading.

18.4.1.1.2 This test is not considered necessary where the candidate for "ammonium nitrate emulsion, suspension or gel, intermediate for blasting explosives" is manufactured at a temperature which is 20 °C, or higher, than the maximum temperature which may occur during transport.

18.4.1.2 *Apparatus and materials*

18.4.1.2.1 The experimental equipment consists of a suitable thermostatically controlled test chamber (which may be fan assisted), appropriate ~~Dewar~~insulated test vessels with closures, temperature probes and ~~measuring~~recording equipment.

18.4.1.2.2 The test should be performed ~~in a test cell capable of withstanding fire and overpressure and, preferably, should be fitted with a pressure relief system e.g. a blow out panel~~following a risk assessment, taking account of the potential for fire and/or explosion in the test chamber, and the application of appropriate control measures for the protection of persons and property. A number of tests may be run concurrently. The recording system should be housed in a separate observation area.

18.4.1.2.3 ~~A thermostatically controlled drying oven (which may be fan assisted)~~The test chamber must be large enough to allow air circulation on all sides of the ~~Dewar~~insulated test vessels. ~~may be used~~. The air temperature in the ~~oven~~test chamber should be controlled so that the desired temperature for a liquid inert sample in the ~~Dewar~~insulated test vessel can be maintained with a deviation of not more than ± 2~~1~~ °C for up to 10 days. The air temperature in the ~~oven~~test chamber should be measured and recorded. ~~It is recommended that the door of the oven be fitted with a magnetic catch or replaced by a loosely fitting insulated cover. The oven may be protected by an appropriate steel liner and the Dewar vessel housed in a wire mesh cage.~~

18.4.1.2.4 ~~Dewar~~Insulated test vessels with a volume of approximately 500 m~~500 ml~~ with a closure system are used. The closure of the ~~Dewar~~test vessel should be inert. ~~A closure system is illustrated in Figure 18.4.1.1.~~

18.4.1.2.5 The heat loss characteristics of the system used, i.e. ~~Dewar~~insulated test vessel and closure, ~~should~~must be established prior to performance of the test. Since the closure system has a significant effect on the heat loss characteristics, these can be adjusted to some extent by varying the closure system. The heat loss characteristics ~~can be~~are determined by measuring the half time of cooling of the vessel filled with an

known inert liquid substance e.g. distilled water. ~~having similar physical properties.~~ The heat loss per unit of mass, L (W/kg.K) ~~can be~~is calculated from the half time of cooling, $t_{1/2}$ (s), and the specific heat, Cp (J/kg.K), of the substance using the formula:

$$L = \ln 2 \times (C_p / t_{1/2})$$

18.4.1.2.6 ~~Dewar~~Insulated test vessels filled with 400 ml of inert substance, with a heat loss of 100 mW/kg.K or less are suitable.

~~18.4.1.2.7 The Dewar vessel shall be filled to about 80% of its capacity. In case of a sample with very high viscosity it may be required to have the sample provided with a shape which just fits into the Dewar vessel. The diameter of such a preshaed sample shall be just under the inner diameter of the Dewar vessel. The hollow lower end of the Dewar vessel may be filled with an inert solid substance prior to loading the sample into the vessel to facilitate the use of cylindrically shaped sample substances.~~

18.4.1.3 Procedure

18.4.1.3.1 Set the test chamber at a temperature which is 20 °C higher than the maximum temperature which may occur during transport or, if higher, at the temperature at the time of loading. Fill the ~~Dewar~~test vessel with the substance under test ~~and note the mass of the sample. Make sure the sample is filled~~ to about 80% of ~~its height~~the capacity of the test vessel, or approximately 400 ml. Insert the temperature probe into the centre of the sample. Seal the lid of the ~~Dewar~~test vessel ~~in place~~ and ~~insert~~place ~~the Dewar vessel~~it in the test chamber, connect the temperature recording system and close the test chamber.

18.4.1.3.2 ~~The sample is heated and t~~The temperature of the sample and of the test chamber are continuously monitored. The time is noted at which the sample temperature reaches a temperature 2 °C below the test chamber temperature. The test is then continued for a further seven days or until the sample temperature rises to 6 °C or more above the test chamber temperature if this occurs sooner. ~~Note the time taken for the sample to rise from 2 °C below the test chamber temperature to its maximum temperature.~~

18.4.1.3.3 ~~If the sample survives~~At the end of the test, allow the sample to cool, ~~and~~ remove it from the test chamber and carefully dispose of it as soon as possible. ~~The percentage mass loss and change in composition may be determined.~~

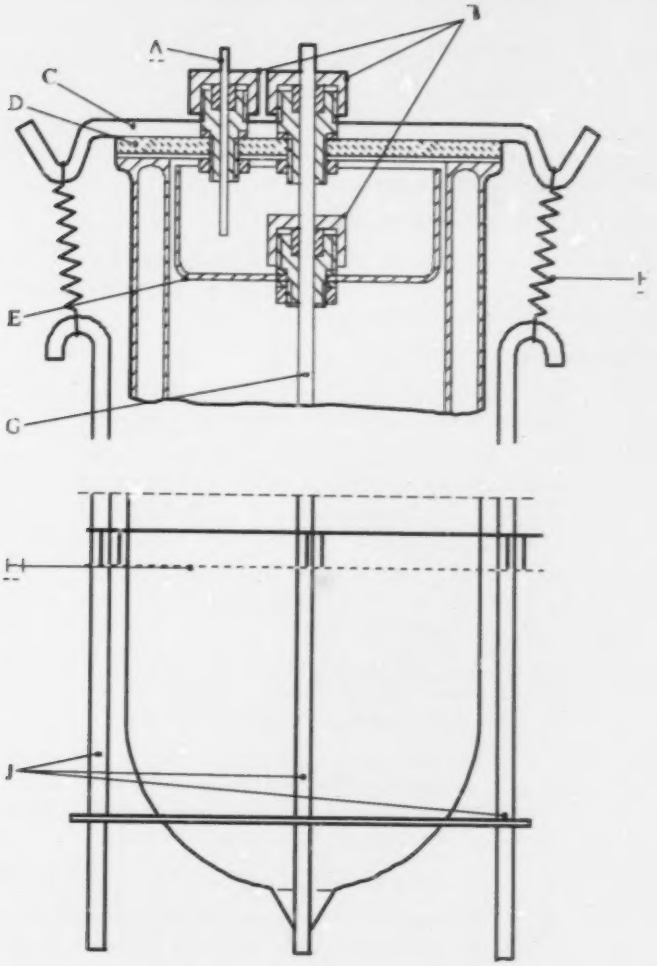
~~18.4.1.3.3~~

18.4.1.4 *Test criteria and method of assessing results*

18.4.1.4.1 If the sample temperature does not exceed the test chamber temperature by 6 °C or more within the seven day period in any test, the ammonium nitrate emulsion, suspension or gel is considered to be thermally stable and can be further tested as a candidate for "ammonium nitrate emulsion, suspension or gel, intermediate for blasting explosives".

18.4.1.5 *Examples of results*

Substances	***Sample mass (g)***	***Test T (°C)***	***Result Comments***
Ammonium nitrate	408	102	- slight discolouration, hardened into lump Mass loss 0.5%
ANE-1 Ammonium nitrate 76%, Water 17%, Fuel/emulsifier 7%	551	102	- separation of oil and crystallized salts. Mass loss 0.8%
ANE-2 (sensitized) Ammonium nitrate 75%, Water 17%, Fuel/emulsifier 7%	501	102	- Some discolouration Mass loss 0.5%
ANE-Y Ammonium nitrate 77%, Water 17%, Fuel/emulsifier 7%	500	85	- Mass loss 0.1%
ANE-Z Ammonium nitrate 75%, Water 20%, Fuel/emulsifier 5%	510	95	- Mass loss 0.2%
ANE-G1 Ammonium nitrate 74%, Sodium nitrate 1%, Water 16%, Fuel/emulsifier 9%	553	85	- no rise in temperature
ANE-G2 Ammonium nitrate 74%, Sodium nitrate 3%, Water 16%, Fuel/emulsifier 7%	540	85	- no rise in temperature
ANE-J1 Ammonium nitrate 80%, Water 13%, Fuel/emulsifier 7%	613	80	- Mass loss 0.1%
ANE-J2 Ammonium nitrate 76%, Water 17%, Fuel/emulsifier 7%	605	80	- Mass loss 0.3%
ANE-J4 Ammonium nitrate 71%, Sodium nitrate 11%, Water 12%,	602	80	- Mass loss 0.1%



~~(A)~~	~~PTFE capillary tube~~	~~(B)~~	~~Special screw fittings (PTFE or Al) with O-ring seal~~
~~(C)~~	~~Metal strip~~	~~(D)~~	~~Glass lid~~
~~(E)~~	~~Glass beaker base~~	~~(F)~~	~~Spring~~
~~(G)~~	~~Glass protective tube~~	~~(H)~~	~~Dewar vessel~~
~~(J)~~	~~Steel retaining device~~		

~~Figure 18.4.1.1: DEWAR VESSEL WITH CLOSURE~~

18.5 Series 8 Type (b) Test prescription

18.5.1 *Test 8 (b): ANE Gap Test*

18.5.1.1 *Introduction*

This test is used to measure the sensitivity of a candidate for "ammonium nitrate emulsion or suspension or gel, intermediate for blasting explosives" to a specified shock level, i.e. a specified ~~donor~~booster charge and gap.

18.5.1.2 *Apparatus and materials*

18.5.1.2.1 The set-up for this test consists of an explosive charge (~~donor~~booster charge), a barrier (gap), a container holding the ~~test charge~~sample substance (acceptor charge), and a steel witness plate (target).

The following materials are to be used:

(a) Detonators of sufficient strength to effectively initiate the booster charge ~~United Nations Standard detonator or equivalent~~;

(b) Booster charges consisting of 95 mm diameter by 95 mm long pellet with a density of 1 600 kg/m³ ± 50 kg/m³ of either ~~50/50~~ pPentolite (PETN/TNT with a minimum 50% PETN), Composition B (RDX/TNT with a minimum 50% RDX) or ~~95/5~~ RDX/WAX (with a minimum 95% RDX);

(c) Tubing, steel, ~~seamless,~~ with an outer diameter of 95.0 ± 7.0 mm, a wall thickness of 9.75 ± 2.75 mm and an inner diameter of 73.0 ± 7.0 mm, and with a length of 280 mm;

(d) Sample substances (acceptor charges); ~~with a diameter which is just under the inner diameter of the steel tubing. The air gap between the sample and tubing wall should be as small as possible;~~

(e) Polymethyl methacrylate (PMMA) rod, of 95 mm diameter by 70 mm long; ~~A gap length of 70 mm results in an incident shock pressure at the ANE interface somewhere between 3.5 and 4 GPa, depending on the type of donor used (see Table 18.5.1.1 and Figure 18.5.1.2);~~

(f) Mild steel plate, approximately 200 mm × 200 mm × 20 mm;

(g) Wood blocks, 95 mm diameter and approximately 25 mm thick, with a hole drilled through the centre to hold the detonator in place against the booster charge;

~~(g)~~(h) Wood blocks or similar to stand the assembly at least 100 mm off the ground.

18.5.1.3 *Procedure*

18.5.1.3.1 As shown in Figure 18.5.1.1, the detonator, ~~donor~~booster charge, PMMA gap and acceptor charge are coaxially aligned above the centre of the witness plate. The bottom end of the tube is sealed with a single layer of cloth adhesive tape, or equivalent, to contain the sample substance which is carefully loaded so as to avoid the formation of voids within the sample or between the sample and the tube walls. The surface of the sample should be level with the rim of the tube. Care should be taken to ensure good contact between the detonator, the booster charge, ~~and donor, donor and gap and~~the PMMA cylinder ~~gap~~ and the acceptor charge. ~~The test sample and booster should be at ambient temperature for the test.~~ The sample substance should be at ambient

temperature or not more than 30°C. The wood block holding the detonator, the booster charge, the PMMA cylinder and the steel tube should be held firmly in alignment (e.g. by using a band of adhesive tape at each intersection).

18.5.1.3.2 ~~To assist in collecting the remains of the witness plate, the whole assembly may be mounted over a container of water with at least a 10 cm air gap between the surface of the water and the bottom surface of the witness plate which should be supported along two edges only.~~ The whole assembly, including the witness plate, is raised above the ground, with at least a 100 mm air gap between the ground and the bottom surface of the witness plate which is supported along two edges only with wooden blocks, or similar, as shown in Figure 18.5.1.1. The location of the blocks must ensure there is a clear space under where the tube is standing on the witness plate. To assist in collecting the remains of the witness plate, the whole assembly should be vertical (e.g. checked with a spirit level).

18.5.1.3.3 ~~Alternative collection methods may be used but it is important to allow sufficient free space below the witness plate so as not to impede plate puncture.~~ The test is performed three times unless a positive result is observed earlier

18.5.1.4 *Test criteria and method of assessing results*

A clean hole punched through the plate indicates that a detonation was initiated and propogated in the sample. A substance which detonates and punches a hole in the witness plate in any trial ~~at a gap length of 70 mm~~ is not to be classified as "ammonium nitrate emulsion or suspension or gel, intermediate for blasting explosives" and the result is noted as "+".

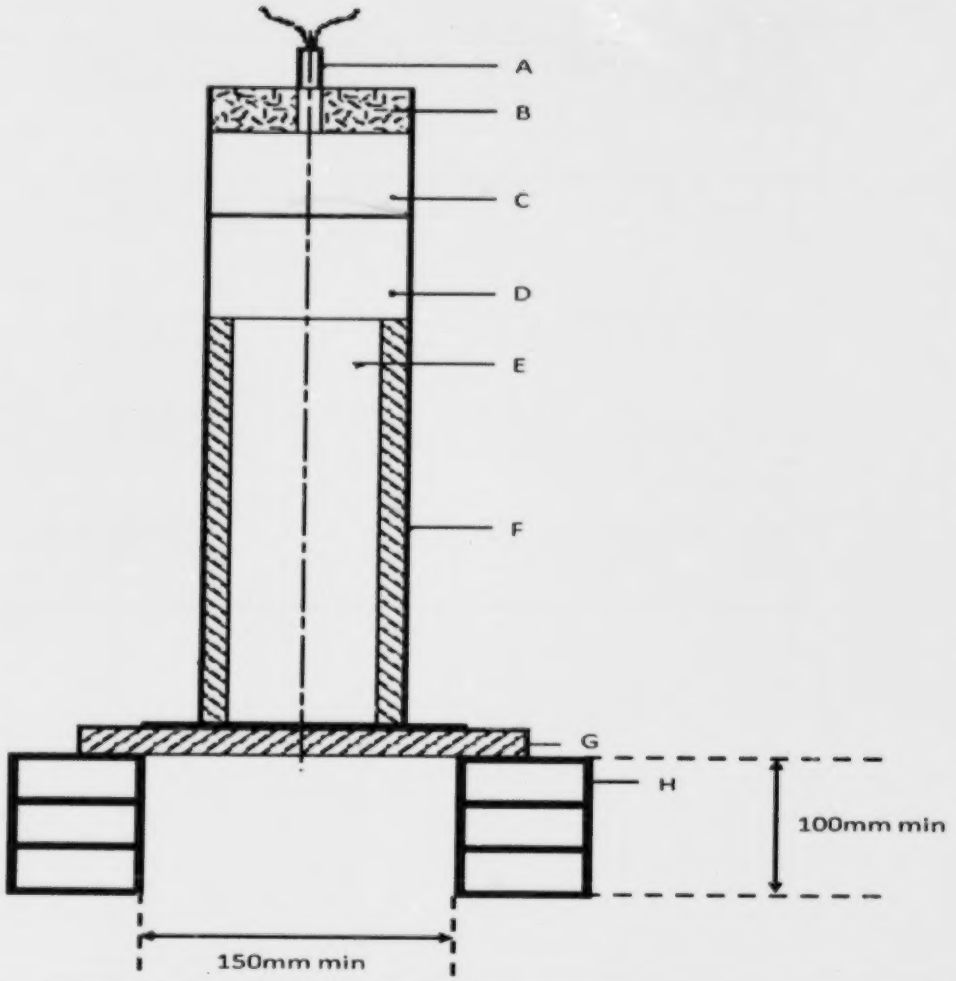
18.5.1.5 *Examples of results*

Substances	*Density g/cm3*	*Gap mm*	*Result*	*Comments*
Ammonium nitrate (low density)	0.85	35	-	Tube fragmented (large fragments) Plate bent VOD 2.3-2.8 km/s
Ammonium nitrate (low density)	0.85	35	-	Tube fragmented (large fragments) Plate fractured
ANE-FA Ammonium nitrate 69%, Sodium nitrate 12%, Water 10%,	1.4	50	-	Tube fragmented (large fragments) Plate not perforated
ANE-FA	1.44	70	-	Tube fragmented (large fragments) Plate not perforated
ANE-FB Ammonium nitrate 70%, Sodium nitrate 11%, Water 12%,	ca 1.40	70	-	Tube fragmented (large fragments) Plate not perforated
ANE-FC (sensitized) Ammonium nitrate 75%, Water 13%, Fuel/emulsifier 10%	1.17	70	+	Tube fragmented (fine fragments) Plate perforated
ANE-FD (sensitized) Ammonium nitrate 76%, Water 17%, Fuel/emulsifier 7%	ca 1.22	70	+	Tube fragmented (fine fragments) Plate perforated

Substances	*Density g/cm3*	*Gap mm*	*Result*	*Comments*
ANE-1 Ammonium nitrate 76%, Water 17%, Fuel/emulsifier 7%	1.4	35	-	Tube fragmented into large pieces. Plate dented VOD: 3.1 km/s
ANE-2 (sensitized) Ammonium nitrate 76%, Water 17%, Fuel/emulsifier 7%	1.3	35	+	Tube fragmented into small pieces Plate perforated VOD: 6.7 km/s

Substances	*Density g/cm3*	*Gap mm*	*Result*	*Comments*
ANE-2 (sensitized) Ammonium nitrate 76%, Water 17%, Fuel/emulsifier 7%	1.3	70	+	Tube fragmented into small pieces Plate perforated VOD: 6.2 km/s
ANE-G1 Ammonium nitrate 74%, Sodium nitrate 1%, Water 16%, Fuel/emulsifier 9%	1.29	70	-	Tube fragmented Plate indented VOD 1968 m/s
ANE-G2 Ammonium nitrate 74%, Sodium nitrate 3%, Water 16%,	1.32	70	-	Tube fragmented Plate indented
ANE-G3 (sensitized by gassing) Ammonium nitrate 74%, Sodium nitrate 1%, Water 16%, Fuel/emulsifier 9%	1.17	70	+	Tube fragmented Plate punctured
ANE-G4 (sensitized by microballoons) Ammonium nitrate 74%, Sodium nitrate 3%, Water 16%, Fuel/emulsifier 7%	1.23	70	+	Tube fragmented Plate punctured
ANE-G5 Ammonium nitrate 70%, Calcium nitrate 8%, Water 16%, Fuel/emulsifier 7%	1.41	70	-	Tube fragmented Plate indented VOD 2 061 m/s

Substances	*Density g/cm3*	*Gap mm*	*Result*	*Comments*
ANE-J1 Ammonium nitrate 80%, Water 13%, Fuel/emulsifier 7%	1.39	70	-	Tube fragmented Plate indented
ANE-J2 Ammonium nitrate 76%, Water 17%, Fuel/emulsifier 7%	1.42	70	-	Tube fragmented Plate indented
ANE-J4 Ammonium nitrate 71%, Sodium nitrate 11%, Water 12%,	1.40	70	-	Tube fragmented Plate indented
ANE-J5 (sensitized by microballoons) Ammonium nitrate 71%, Sodium nitrate 5%, Water 18%,	1.20	70	+	Tube fragmented Plate perforated VOD 5.7 km/s
ANE-J6 (sensitized by microballoons) Ammonium nitrate 80%, Water 13%,	1.26	70	+	Tube fragmented Plate perforated VOD 6.3 km/s



(A) Detonator (B) Wooden detonator holder (C) Booster charge

(D) PMMA gap (E) Substance under test (F) Steel Tube

(G) Witness plate (H) Wooden blocks

Figure 18.5.1.1: ANE GAP TEST

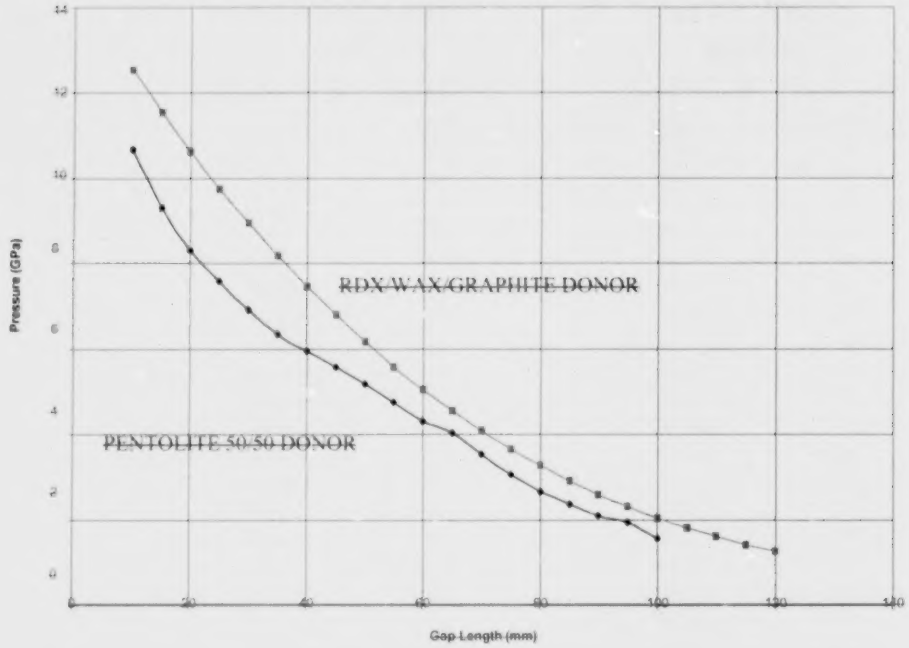
~~(A) Detonator~~ ~~(B) Booster charge~~
~~(C) PMMA gap~~ ~~(D) Substance under test~~
~~(E) Steel tube~~ ~~(F) Witness plate~~

~~**Figure 18.5.1.1: ANE GAP TEST**~~

~~Table 18.5.1.1: ANE GAP TEST CALIBRATION DATA~~

~~PENTOLITE 50/50 DONOR~~		~~RDX/WAX/GRAPHITE DONOR~~	
~~Gap length (mm)~~	~~Barrier pressure (GPa)~~	~~Gap length (mm)~~	~~Barrier pressure (GPa)~~
~~10~~	~~10.67~~	~~10~~	~~12.53~~
~~15~~	~~9.31~~	~~15~~	~~11.55~~
~~20~~	~~8.31~~	~~20~~	~~10.63~~
~~25~~	~~7.58~~	~~25~~	~~9.76~~
~~30~~	~~6.91~~	~~30~~	~~8.94~~
~~35~~	~~6.34~~	~~35~~	~~8.18~~
~~40~~	~~5.94~~	~~40~~	~~7.46~~
~~45~~	~~5.56~~	~~45~~	~~6.79~~
~~50~~	~~5.18~~	~~50~~	~~6.16~~
~~55~~	~~4.91~~	~~55~~	~~5.58~~
~~60~~	~~4.51~~	~~60~~	~~5.04~~
~~65~~	~~4.02~~	~~65~~	~~4.54~~
~~70~~	~~3.53~~	~~70~~	~~4.08~~
~~75~~	~~3.05~~	~~75~~	~~3.66~~
~~80~~	~~2.66~~	~~80~~	~~3.27~~
~~85~~	~~2.36~~	~~85~~	~~2.91~~
~~90~~	~~2.10~~	~~90~~	~~2.59~~
~~95~~	~~1.94~~	~~95~~	~~2.31~~
~~100~~	~~1.57~~	~~100~~	~~2.04~~
		~~105~~	~~1.81~~
		~~110~~	~~1.61~~
		~~115~~	~~1.42~~
		~~120~~	~~1.27~~

Figure 18.5.1.2: ANE Gap Test Calibration Data



18.6 Series 8 Type (c) Test prescription

18.6.1 Test 8 (c): Koenen test

18.6.1.1 *Introduction*

This test is used to determine the sensitiveness of a candidate ammonium nitrate emulsion or suspension or gel, intermediate for blasting explosive, to the effect of intense heat under high confinement.

18.6.1.2 *Apparatus and materials*

18.6.1.2.1 The apparatus consists of a non-reusable steel tube, with its re-usable closing device, installed in a heating and protective device. The tube is deep drawn from sheet steel conforming to specification DC04 (EN 10027-1), or equivalent A620 (AISI/SAE/ASTM), or equivalent SPCEN (JIS G 3141). The dimensions are given in Figure 18.6.1.1. The open end of the tube is flanged. The closing plate with an orifice, through which the gases from the decomposition of the test substance escape, is made from heat-resisting chrome steel and is available with the following diameter holes:

- 1.5 mm for the closing plate used in the heating calibration procedure; and
- 2.0 mm for the closing plate used in the test.

~~18.6.1.2.1 1.0 1.5 2.0 2.5 3.1 5.0 8.0 12.0 20.0 mm.~~ The dimensions of the threaded collar and the nut (closing device) are given in Figure 18.6.1.1.

For quality control of the steel tubes, 1% of the tubes from each production lot shall be subjected to quality control and the following data shall be verified:

(a) The mass of the tubes shall be 26.5 ± 1.5 g~~, tubes to be used in one test sequence shall not differ in mass by more than 1 g~~;

(b) The length of the tubes shall be 75 ± 0.5 mm;

(c) The wall thickness of the tubes measured 20 mm from the bottom of the tube shall be 0.5 ± 0.05 mm; and

(d) The bursting pressure as determined by quasi-static load through an incompressible fluid shall be 30 ± 3 MPa.

18.6.1.2.2 Heating is provided by a gaseous fuel (e.g. propane), from an industrial cylinder fitted with a pressure regulator, via a flow meter and distributed by a manifold to the four burners. ~~Other fuel gases may be used providing the specified heating rate is obtained.~~ The gas pressure is regulated to give a heating rate of 3.3 ± 0.3 K/s when measured by the calibration procedure. Calibration involves heating a tube (fitted with a 1.5 mm orifice plate) filled with 27 cm^3 of dibutyl phthalate. The time taken for the temperature of the liquid (measured with a 1 mm diameter thermocouple centrally placed 43 mm below the rim of the tube and inserted through the orifice plate) to rise from 135 °C to 285 °C is recorded and the heating rate calculated.

18.6.1.2.3 Because the tube is likely to be destroyed in the test, heating is undertaken in a protective welded box~~,~~ A suitable arrangement of the construction and dimensions of the box ~~which are~~is given in Figure 18.6.1.2. The tube is suspended between two rods placed through holes drilled in opposite walls of the box. ~~The~~A suitable arrangement of the burners is given in Figure 18.6.1.2. The burners are lit simultaneously by a pilot flame or an electrical ignition device. ***The test apparatus is placed in a protective area***. Measures should be taken to ensure that any draughts does not affect the burner

flames. Provision should be made for extracting any gases or smoke resulting from the test.

~~18.6.1.2.3~~ 18.6.1.2.4 A video camera should be provided to record the test and to ensure all burners are functional during the test. The cameras may also provide evidence of blockages of the orifice by solids within the sample.

18.6.1.3 Procedure

18.6.1.3.1 The substance is loaded into the tube to a height of 60 mm taking particular care to prevent the formation of voids. The threaded collar is slipped onto the tube from below, the ~~appropriate~~2 mm orifice plate is inserted and the nut tightened by hand after applying some high temperature anti-seize compound (e.g. molybdenum disulphide based lubricant). It is essential to check that none of the substance is trapped between the flange and the plate, or in the threads.

18.6.1.3.2 ~~With orifice plates from 1.0 mm to 8.0 mm diameter, nuts with an orifice of 10.0 mm diameter should be used; if the diameter of the orifice is above 8.0 mm, that of the nut should be 20.0 mm.~~ Each tube is used for one trial only. The orifice plates, threaded collars and nuts may be used again provided they are undamaged.

18.6.1.3.3 The tube is placed in a rigidly mounted vice and the nut tightened with a spanner. The tube is then suspended between the two rods in the protective box. The test area is vacated, the gas supply turned on and the burners lit. ~~The time to reaction and duration of reaction can provide additional information useful in interpreting the results.~~ If rupture of the tube does not occur, heating is to be continued for at least five minutes before the trial is finished. After each trial the fragments of the tube, if any, should be collected and weighed to ensure all pieces have been recovered.

18.6.1.3.4 The following effects are differentiated:

"O": Tube unchanged;
"A": Bottom of tube bulged out;
"B": Bottom and wall of the tube bulged out;
"C": Bottom of tube split;
"D": Wall of tube split;
"E": Tube split into two[1] fragments;
"F": Tube fragmented into three[1] or more mainly large pieces which in some cases may be connected with each other by a narrow strip;
"G": Tube fragmented into many mainly small pieces, closing device undamaged; and
"H": Tube fragmented into many very small pieces, closing device bulged out or fragmented.

Examples for the effect types "D", "E" and "F" are shown in Figure 18.6.1.3. If a trial results in any of the effects "O" to "E", the result is regarded as "no explosion". If a trial gives the effect "F", "G" or "H", the result is evaluated as "explosion".

18.6.1.3.5 The test is performed to achieve negative (-) results in three tests.

Given the nature of ammonium nitrate emulsions, suspensions or gels and the possibility of varying percentages of solids present, blockages of the orifices may occur during testing potentially leading to a false "+" result. Where this is observed the test may be repeated (maximum twice).~~series of trials is started with a single trial using an orifice plate of 20.0 mm. If, in this trial, the result "explosion" is observed, the series is continued with trials using tubes without orifice plates and nuts but with threaded collars (orifice 24.0 mm). If at 20.0 mm "no explosion" occurs, the series is continued with single trials using plates with the following orifices 12.0 – 8.0 – 5.0 – 3.0 – 2.0 – 1.5 and finally 1.0 mm~~

~~until, at one of these diameters, the result "explosion" is obtained. Subsequently, trials are carried out at increasing diameters, according to the sequence given in 18.6.1.2.1, until only negative results in three tests at the same level are obtained. The limiting diameter of a substance is the largest diameter of the orifice at which the result "explosion" is obtained. If no "explosion" is obtained with a diameter of 1.0 mm, the limiting diameter is recorded as being less than 1.0 mm.~~

18.6.1.4 Test criteria and method of assessing results

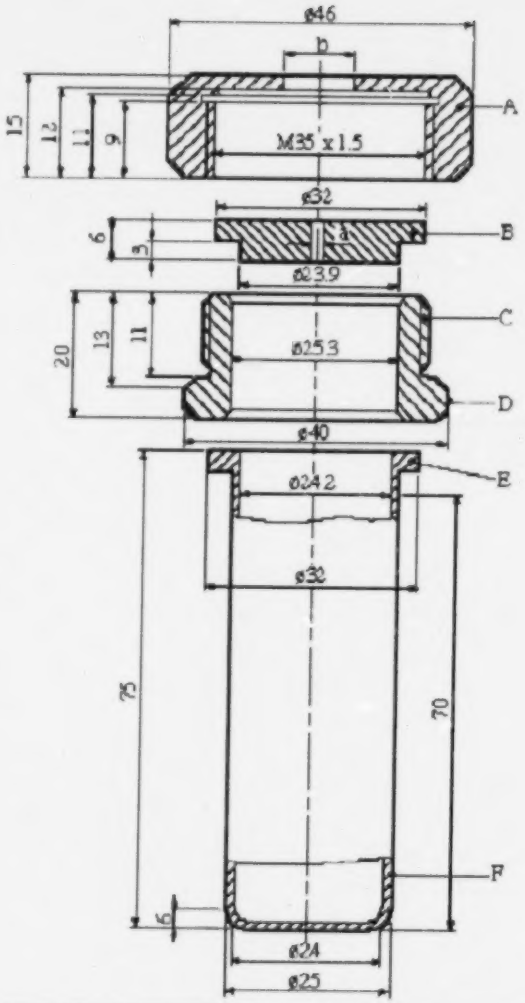
The result is considered "+" and the substance should not be classified in Division 5.1 if th<u>ree negative(-) results cannot be achieved within a maximum of five tests.</u> ~~e limiting diameter is 2.0 mm or more. The result is considered "-" if the limiting diameter is less than 2.0 mm.~~

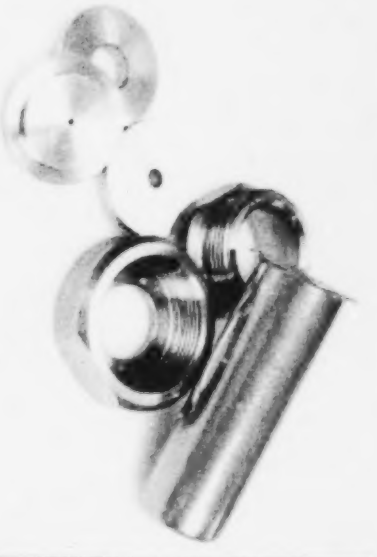
[1] The upper part of the tube remaining in the closing device is counted as one fragment.

18.6.1.5 *Examples of results*

Substances	*Result*	*Comments*
Ammonium nitrate (low density)	–	Limiting diameter: <1 mm
ANE-F1 Ammonium nitrate 71%, Water 21%, Fuel/emulsifier 7%	–	
ANE-F2 Ammonium nitrate 77%, Water 17%, Fuel/emulsifier 7%	–	
ANE-F3 Ammonium nitrate 70%, Sodium nitrate 11%, Water 12%, Fuel/emulsifier 7%	–	
ANE-F4 Ammonium nitrate 42%, Calcium nitrate 35%, Water 16%, Fuel/emulsifier 7%	–	
ANE-F5 Ammonium nitrate 69%, Sodium nitrate 13%, Water 10%, Fuel/emulsifier 8%	–	
ANE-F6 Ammonium nitrate 72%, Sodium nitrate 11%, Water 10%, Fuel/emulsifier 6%	–	
ANE-F7 Ammonium nitrate 76%, Water 13%, Fuel/emulsifier 10%	–	
ANE-F8 Ammonium nitrate 77%, Water 16%, Fuel/emulsifier 6%	–	
ANE-1 Ammonium nitrate 76%, Water 17%, Fuel/emulsifier 7%	–	Limiting diameter: 1.5 mm
ANE-2 (sensitized by microballoons) Ammonium nitrate 75%, Water 17%, Fuel/emulsifier 7%	–	Limiting diameter: 2 mm
ANE-4 (sensitized by microballoons) Ammonium nitrate 70%, Sodium nitrate 11%, Water 9%, Fuel/emulsifier 5.5%	–	Limiting diameter: 2 mm
ANE-G1 Ammonium nitrate 74%, Sodium nitrate 1%, Water 16%, Fuel/emulsifier 9%	–	

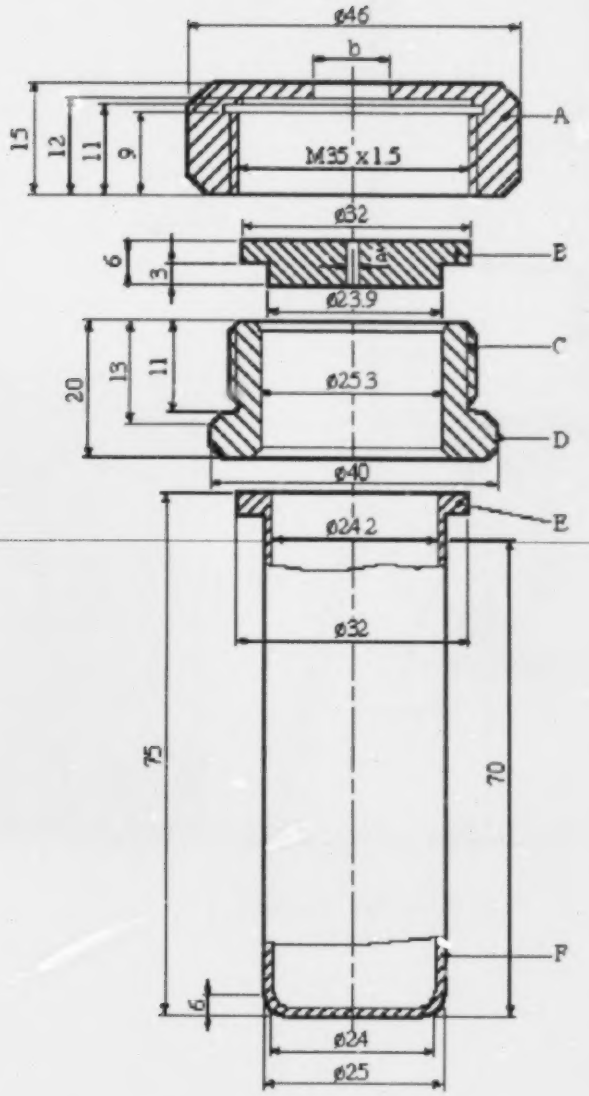
Substances	*Result*	*Comments*
ANE-G2 Ammonium nitrate 74%, Sodium nitrate 3%, Water 16%, Fuel/emulsifier 7%		
ANE-J1 Ammonium nitrate 80%, Water 13%, Fuel/emulsifier 7%		Effect type "O"
ANE-J2 Ammonium nitrate 76%, Water 17%, Fuel/emulsifier 7%		Effect type "O"
ANE-J4 Ammonium nitrate 71%, Sodium nitrate 11%, Water 12%, Fuel/emulsifier 6%		Effect type "A"





(A) Nut (b = 10 mm) with flats for size 41 spanner
(B) Orifice plate (a = 1.5 or 2.0 mm)
(C) Threaded collar
(D) Flats for size 36 spanner
(E) Flange
(F) Tube

Figure 18.6.1.1: Test Tube Assembly



(A) Nut (b = 10.0 or 20.0 mm) with flats for size 41 spanner
(B) Threaded collar
(C) Flange
(A) Orifice plate (a = 1.0 to 20.0 mm diameter)
(D) Flats for size 36 spanner
(F) Tube

Figure 18.6.1.1: TEST TUBE ASSEMBLY

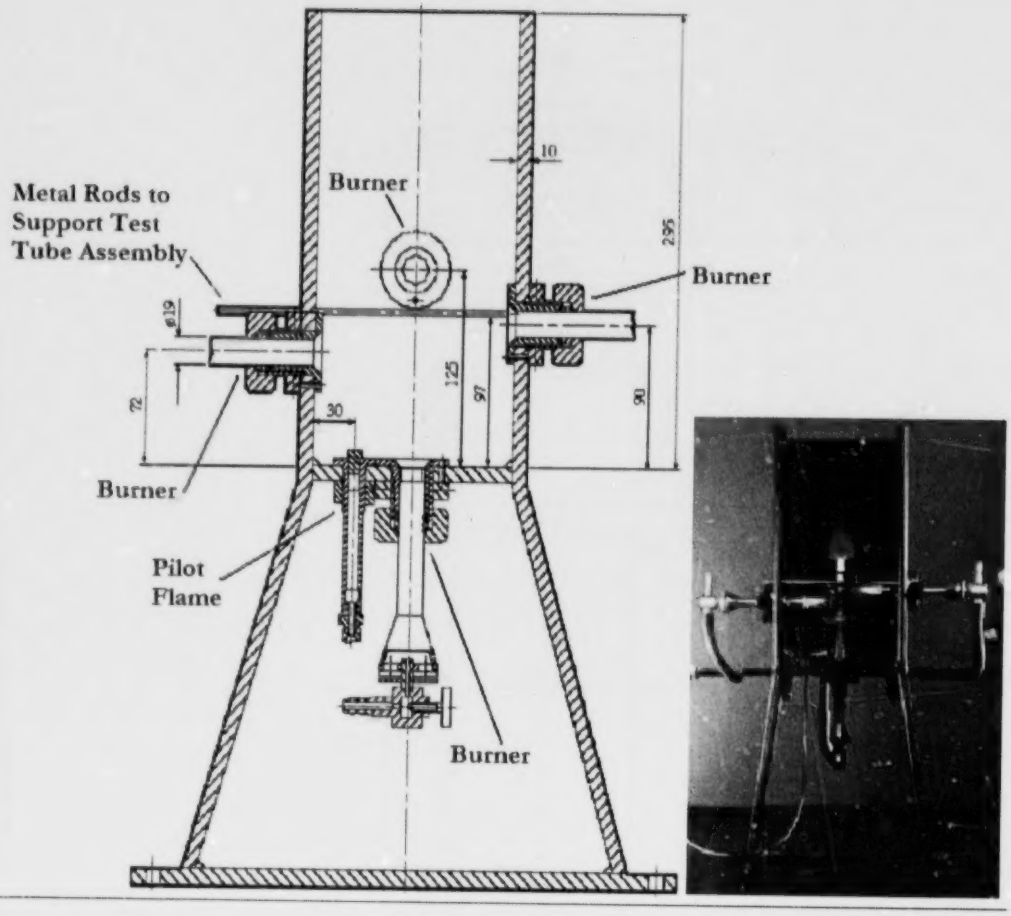
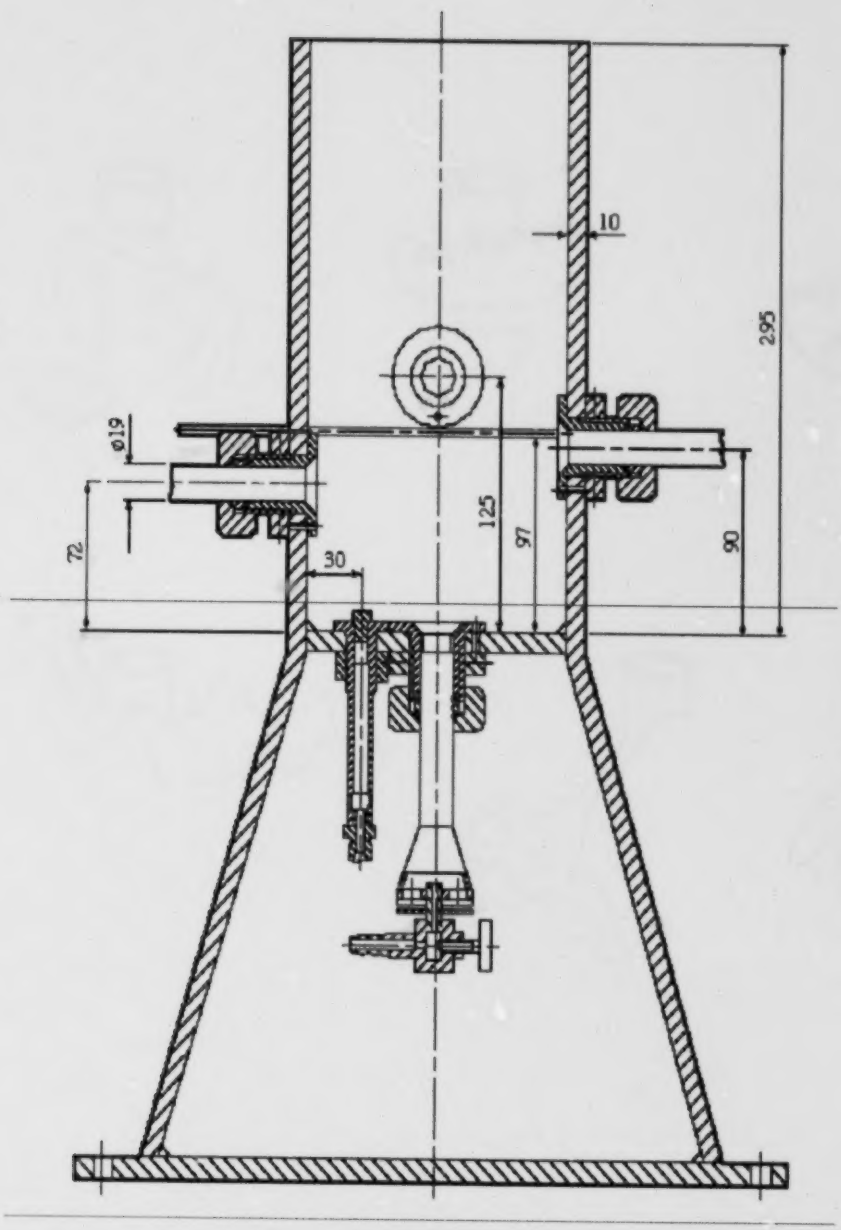


Figure 18.6.1.2 Heating Device



~~Figure 18.6.1.2: HEATING AND PROTECTIVE DEVICE~~

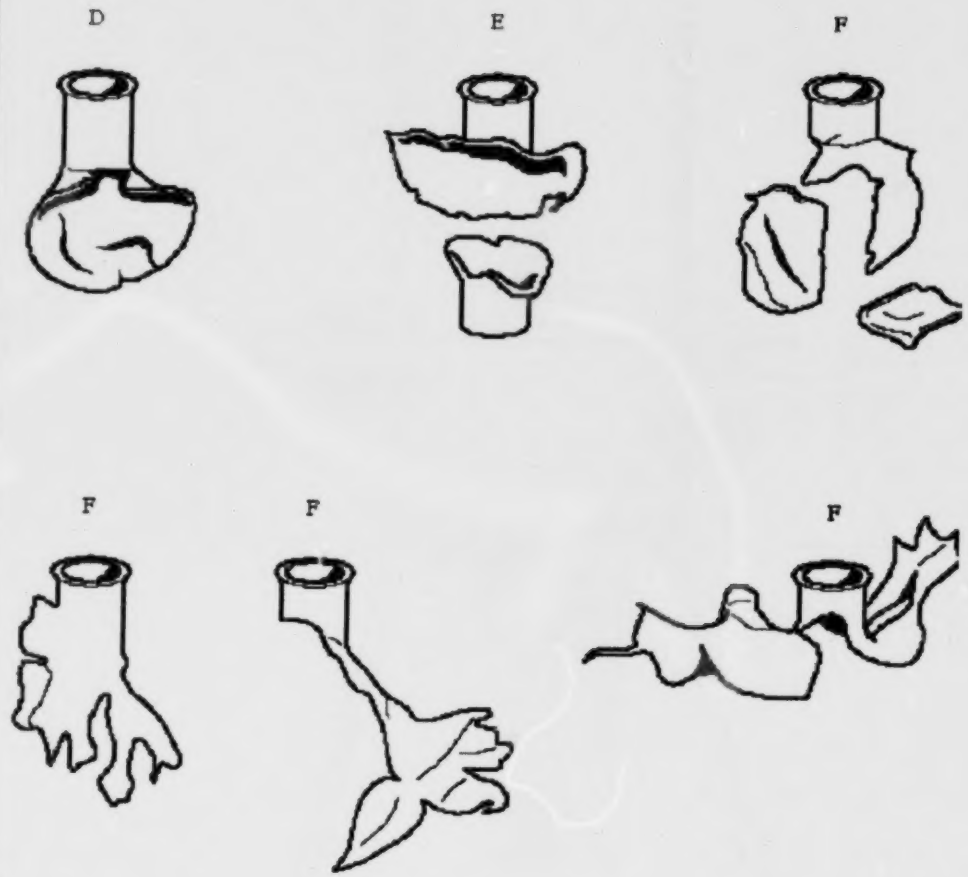


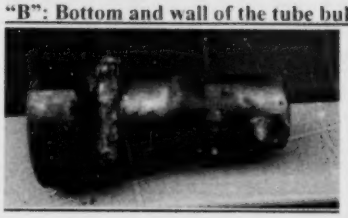
Figure 18.6.1.3: EXAMPLES OF EFFECT TYPES D, E AND F

Examples of Koenen test results

"O": Tube unchanged

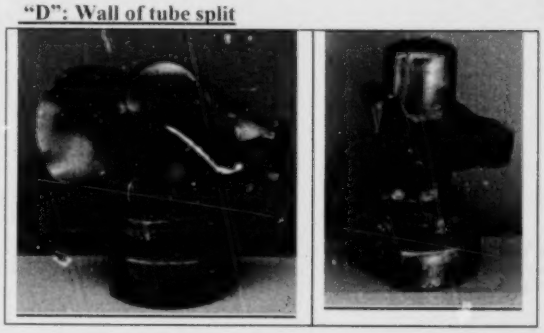
"A": Bottom of the tube bulged out

"B": Bottom and wall of the tube bulged out



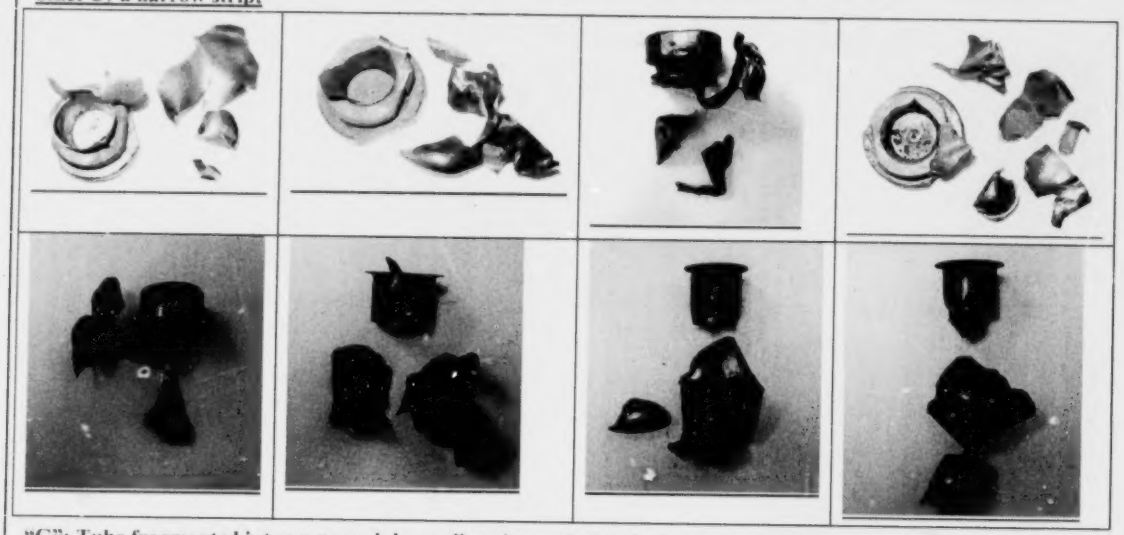
Classification "C": Bottom of tube split

"D": Wall of tube split

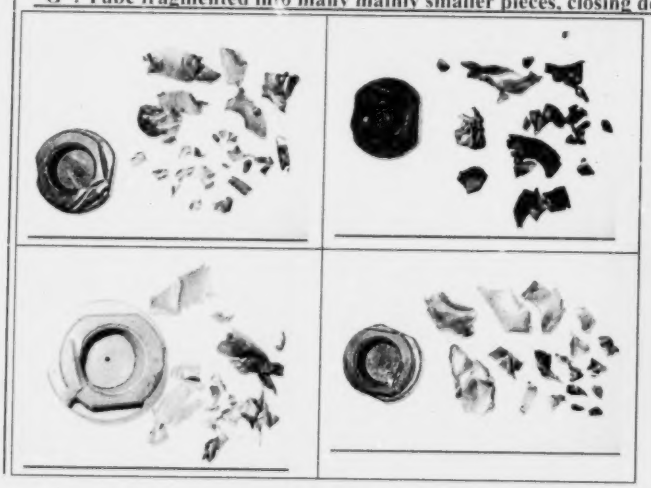


"E": Tube split into two fragments

"F": Tube fragmented into three or more mainly large pieces which in some cases may be connected with each other by a narrow strip;



"G": Tube fragmented into many mainly smaller pieces, closing device undamaged



~~18.7 Series 8 Type (d) Test prescriptions~~

~~18.7.1~~

18.7 Series 8 Type (d) Test prescriptions

~~18.7.2~~ 18.7.1 Test 8 (d) (i): Vented pipe test

~~18.7.2.1~~18.7.1.1 Introduction

This test is not intended for classification but is included in this Manual for evaluating the suitability of a candidate for "ammonium nitrate emulsion or suspension or gel, intermediate for blasting explosives" for transport in portable tanks as a dangerous substance of Division 5.1.

Where this test is undertaken, test 8(c) may be omitted.

The vented pipe test is used to assess the effect of exposure of a candidate for "ammonium nitrate emulsion or suspension or gel, intermediate for blasting explosives" to a large fire under confined, vented conditions.

~~18.7.2.2~~18.7.1.2 Apparatus and materials

The following items are needed:

(a) A steel pipe 310 ± 10 mm diameter and 610 ± 10 mm long, welded close at the bottom with a 380 mm square, 10 ± 0.5 mm thick mild steel plate. The top of the pipe is welded to a 380 mm square, 10 ± 0.5 mm thick mild steel plate that contains a 78 mm diameter vent hole centrally located in the plate to which a 152 mm long steel pipe nipple of 78 mm internal diameter is welded (see Figure 18.7.1.1). All welding should be to a relevant ISO standard or equivalent. All steel components are to be Schedule 40 carbon steel (A53 Grade B) or equivalent;

(b) A metal grid to support the filled pipe above the ~~fuel~~fire and allow adequate heating. If a wooden crib fire is used, the grid should be approximately 1.0 m above the ground and if a liquid hydrocarbon pool fire is used then the grid ~~shall~~should be approximately 0.5 m above the ground;

(c) Enough fuel to produce a fire reaching 800°C (measured at the external base of the pipe) and to keep ~~a fire~~ burning for at least 30 minutes or, if necessary, until the substance has clearly had enough time to react to the fire, evidenced by ejection of material, smoke, fumes, flames, etc., from the top of the pipe. Variation of temperature below 800°C is normal and should not render the test invalid;

(d) Suitable means of ignition to ignite the fuel from two sides e.g. for a wood fire, kerosene to soak the wood and ~~pyrotechnic~~ igniters ~~with wood wool~~;

(e) Cine or video cameras~~, preferably high speed and normal speed,~~ to record events in colour;

(f) ~~Blast gauges, radiometers and associated recording equipment may also be used~~Means of measuring and recording temperature, up to and above 800°C, with a thermocouple located at the external base of the pipe;

~~(f)~~(g) A means of measuring wind speed, such as an anemometer.

~~18.7.2.3~~18.7.1.3 Procedure

18.7.1.3.1 The pipe is filled with the substance under test without tamping during loading. The substance is carefully packed to prevent adding voids. The steel pipe is placed vertically on the grid and secured from tipping over. Fuel is placed beneath the grid, extending in every direction beyond the pipe so that the fire will fully engulf the pipe. Precautions against side winds may be required to avoid dissipation of the heat. Suitable methods of heating include a wood fire ~~using a lattice of wooden laths~~, a liquid or gas fuel fire that produces a flame temperature of at least 800°C. ~~least 800 °C.~~

18.7.1.3.2 The test should not be performed under conditions where the wind speed consistently exceeds 6 m/s. ~~One method is to use a wood fire which has a balanced air/fuel ratio, thereby avoiding too much smoke which would obscure the events, and which burns with sufficient intensity and duration to bring the substance to a possible reaction. A suitable method involves using air-dried pieces of wood (approximately 50 mm square section), stacked to form a lattice beneath the grid (1 m off the ground), and up to the base of the grid supporting the pipe. The wood should extend beyond the pipe to a distance of at least 1.0 m in every direction and the lateral distance between the laths should be about 100 mm.~~

~~18.7.1.3.3 A receptacle filled with suitable liquid fuel, a combination of both wood and liquid fuel fire may be used as an alternative to the wood fire providing it is as severe. If a liquid pool fire is used, the receptacle should extend beyond the pipe to a distance of at least 1.0 m in every direction. The distance between the grid platform and the receptacle should be approximately 0.5 m. Before using this method, consideration should be given to whether any quenching action or adverse interaction between the substance and the liquid fuel can occur such as might bring the results into question~~

18.7.1.3.3 Observations are made on the following:

(a) Wind speed at commencement of the test as per Section 18.7.1.3.2;

(b) Fire duration of at least 30 minutes or until the substance has clearly had enough time to react to the fire, with 800°C reached at the external base of the pipe;

(c) Temperature at the external base of pipe;

(d) Substance reacting to the fire as described in 18.7.1.2(c);

(e) Evidence of explosion (e.g. fragmentation of the pipe into two or more pieces) within 30 minutes of the fire reaching 800°C at the base of the pipe;

(f) Projection of fragments of the cylindrical pipe section from the fire area within 30 minutes of the fire reaching 800°C at the external base of the pipe;

(g) Evidence of a rupture (e.g. a split of the pipe or separation of the pipe from the base plate at the weld) within 30 minutes of the fire reaching 800°C at the base of the pipe;

~~18.7.1.3.4 If gas is to be used as a fuel, the burning area must extend beyond the pipe to a distance of 1.0 m in every direction. The gas must be supplied in such a manner to ensure that the fire is evenly distributed around the pipe. The gas reservoir should be large enough to keep the fire burning for at least 30 minutes. Ignition of the gas may be accomplished either by remotely ignited pyrotechnics or by remote release of the gas adjacent to a pre-existing source of ignition.~~

~~18.7.1.3.5 The ignition system should be put into place and the fuel ignited on two sides, one up wind, simultaneously. The test should not be performed under conditions where the wind speed exceeds 6 m/s.~~ ***~~The fire shall be started from a safe place. If the pipe does not rupture, the system should be allowed to cool down before carefully dismantling the test set-up and emptying the pipe~~***

~~18.7.1.3.6 Observations are made on the following:~~

~~(a) Evidence of explosion~~

~~(b) Loud noise; and~~

~~(c) Projection of fragments from the fire area.~~

18.7.1.4 Safety issues

18.7.1.4.1 Suitable test site selection criteria

(a) a recommended minimum safety distance of 1km radius from the test area if the test is conducted unprotected;

(b) sufficient distance from public or private areas to eliminate the risk of public exposure to noise, fume and shrapnel;

(c) remote and/or secure enough to prevent unauthorised entry;

(d) minimal significant fire risk in the event of a positive result;

(e) minimal risk from loss of containment issues;

(f) adequate capacity for safe storage of test products; and

(g) appropriate approvals, notifications and awareness.

(h) Multiple escape routes available to test personnel in case of wind-blown fume clouds or bushfires.

18.7.1.4.2 Test precautions

(a) If line of sight to the test is not possible, a remote observation system (e.g. video surveillance) should be used to observe the test from a safe distance;

(b) Re-entry time to the test area should be set at a recommended minimum of 150 minutes if the test remains active;

(c) Appropriate personal protective equipment (PPE) should be provided. Precautions should be taken to minimise risk of exposure to elements, fire, hot objects, fumes, etc.;

(d) Risk of vehicle damage due to shrapnel on or protruding from the ground during re-entry to the test area.

(e) If the pipe does not rupture, the system should be allowed to cool down before carefully dismantling the test set-up and emptying the pipe. Ideally this should be left overnight.

~~18.7.1.4~~ 18.7.1.5 **Test criteria and method of assessing results**

A test is considered valid if observation criteria outlined in Section 18.7.1.3.3 (a) to (d) have been met.

The test result is considered "+" and the substance should not be transported in portable tanks as a dangerous substance of Division 5.1 if an explosion and/or fragmentation of the cylindrical **pipe**, as specified in Section 18.7.1.3.3 (e) and (f) is

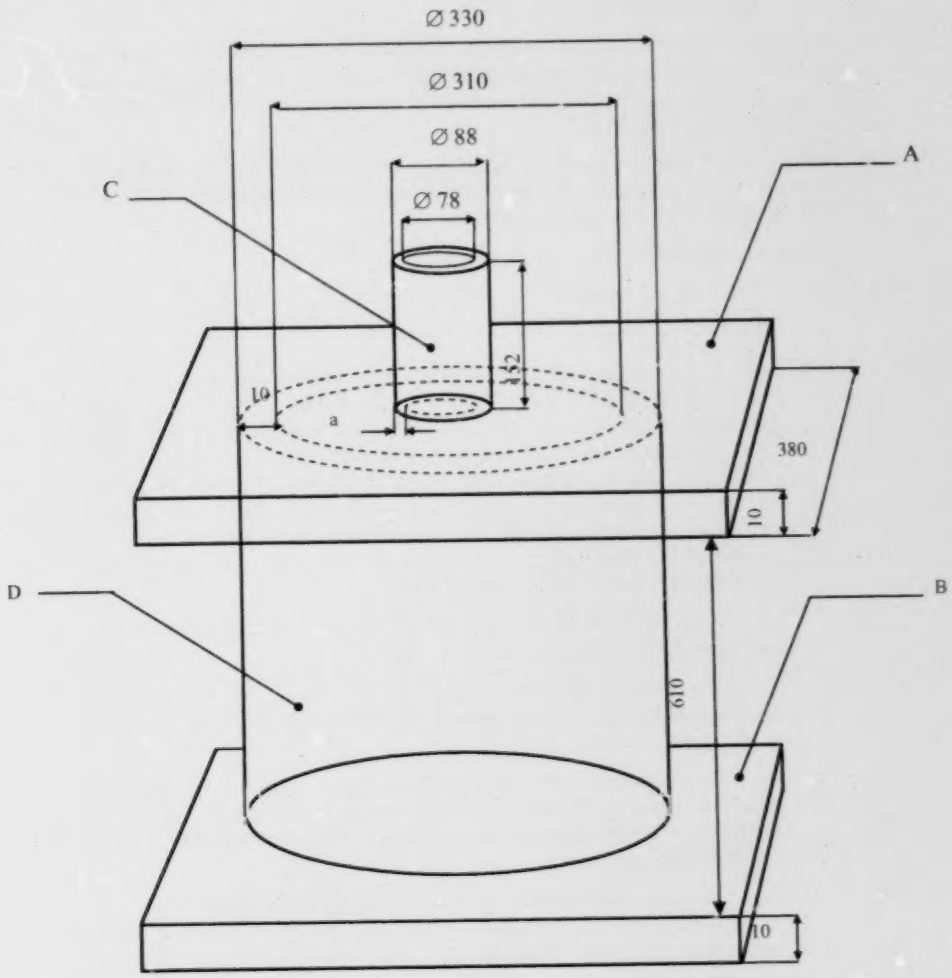
observed within 30 minutes of the fire reaching 800°C at the external base of the pipe. ~~If no explosion and/or fragmentation of the pipe is observed then the re~~

The test ~~sult~~ is considered "-" if no explosion and/or fragmentation of the cylindrical pipe into two or more pieces is observed within 30 minutes of the fire reaching 800°C at the external base of the pipe. Splitting of the cylindrical pipe or its separation from the end plates, as specified in Section 18.7.1.3.3 (g) is evidence of a "-" result.

~~18.7.1.5~~ 18.7.1.6

Examples of results ~~of results~~

Substance	*Result*
To be added	



All measurements are in millimetres

(A) Top plate (Schedule 40 carbon (A53 grade B)
(B) Bottom plate (Schedule 40 carbon (A53 grade B)
(C) Steel pipe nipple (a = 0.5 cm), Schedule 40 carbon (A53 grade B)
(D) Steel pipe (Schedule 40 carbon (A53 grade B))

Figure 18.7.1.1: VENTED PIPE TEST

18.7.2 Test 8 (d) (ii): Modified vented pipe test

18.7.2.1 Introduction

This test is not intended for classification but is included in this Manual for evaluating the suitability of a candidate for "ammonium nitrate emulsion or suspension or gel, intermediate for blasting explosives", bulk substances to be transported in portable tanks as a dangerous substance of Division 5.1.

The modified vented pipe test is used to assess the effect of exposure of a candidate for "ammonium nitrate emulsion or suspension or gel, intermediate for blasting explosives" to a large fire under confined, vented conditions.

Where this test is undertaken, test 8(c) may be omitted.

18.7.2.2 Apparatus and materials

The following items are needed:

(a) A vented vessel consisting of mild drawn steel pipe with an inner diameter of 265 ± 10 mm, a length of 580 ± 10 mm and a wall thickness of 5.0 ± 0.5 mm. Both the top and the base plates are made from 300 mm square, 6.0 ± 0.5 mm thick mild steel plates. The top and base plates are fixed to the pipe with a fillet weld with a thickness of at least 5 mm. All welding should be to a relevant ISO standard or equivalent. The top plate has a vent diameter of 85 mm ± 1.0 mm. A further two small holes are drilled in the top plate to neatly accommodate neatly thermocouple probes;

(b) A concrete block, or similar solid base, about 400 mm square and 50 to 75 mm thick;

(c) A metal stand for supporting the vessel at a height of approximately 150 mm above the concrete block or similar solid base;

(d) A gas burner capable of accommodating a fuel gas (e.g. propane) flow rate of up to 60 g/min. This rests on the concrete block, or similar solid base, under the stand. A typical example of a suitable burner is a 32-jet Mongolian wok burner;

(e) A sheet metal shield to protect the propane flame from side winds. This can be fabricated from approximately 0.5 mm thick galvanised sheet metal. The diameter of the wind shield is about 600 mm and the height isshould be about 250 mm. Four adjustable vents approximately 150 mm wide and 100 mm high are spaced equally around the shield to ensure adequate air reaches the gas flame;

(f) PropaneFuel gas bottle(s) connected via a manifold and fed into a pressure regulator. Other fuel gasas may be used providing the specified heating rate is obtained. The pressure regulator should reduce the propane bottlefuel gas bottle pressure from 600 kPa down to about 150 kPa. The gas then flows through a gas rotameter capable of measuring up to 60 g/min of propane and a needle valve. An electrical solenoid valve is used to switch the propanefuel gas flow on and off remotely. Typically three 9 kg propanefuel gas bottles will achieve the desired gas flow rate for the duration of up to five tests. The gas pressure and flow are regulated to give a heating rate of 3.3 ± 0.3 K/min when measured by the calibration procedure;

(g) Three thermocouples with approximately 500 (2) and 100 (1) mm long stainless steel probes and fiber-glass coated lead wires;

(h) A data-logger capable of recording the output from the thermocouples;

(i) Cine-cameras or video cameras, preferably high speed and normal speed, to record events in colour;

(j) Pure water for calibration;

(k) The candidate ammonium nitrate emulsion or suspension or gel, intermediate for blasting explosives~~ANE~~ to be tested;

(l) A means of measuring wind speed at the commencement of the test, such as an anemometer;

(k)(m) Blast gauges, radiometers and other recording equipment may also be used.

~~Blast gauges, radiometers and associated recording equipment may also be used.~~

18.7.2.3 Calibration

18.7.2.3.1 The vessel is filled to the 75% level (i.e. to a depth of about 435 mm) with the pure water, and heated using the procedure specified in 18.7.2.4. Water is heated from ambient temperature up to 90 °C, monitoring temperature by the thermocouple in the water. Temperature-time data must fit a straight line whose slope will be the "calibration heating rate" for the given combination of vessel and heat source.

18.7.2.3.2 The gas pressure and flow must be regulated to give a heating rate of 3.3 ± 0.3 K/min.

18.7.2.3.3 This calibration must be performed prior to the testing of any ~~ANE~~test substance, though the same calibration can be applied to any test conducted within a day of the calibration provided no change is made to the vessel construction or gas supply. New calibration has to be made every time that the burner is changed.

18.7.2.4 Procedure

18.7.2.4.1 The concrete block, or similar solid base, is placed on a sandy base and levelled using a spirit level. The ~~propane~~fuel gas burner is positioned in the centre of the ~~concrete block~~solid base and connected to the gas supply line. The metal stand is placed over the burner.

18.7.2.4.2 The vessel is placed vertically on the stand and secured from tipping over. The vessel is filled to 75% of its volume (to a height of approximately 435 mm) with the ~~ANE~~substance under test without tamping during loading. The initial temperature of the ~~ANE~~substance must be recorded. The substance is carefully packed to prevent adding voids. The wind shield is positioned around the base of the assembly to protect the ~~propane~~gas flame from heat dissipation due to side winds.

18.7.2.4.3 The thermocouple positions are as follows:

(a) The first 500 mm long probe (T1) in the gas flame;

(b) The second 500 mm long probe (T2) extending all the way into the vessel so that the tip is positioned 80 to 90 mm from the bottom of the vessel;

(c) The third 100 mm long probe (T3) in the headspace about 20 mm into the vessel.

The thermocouples are connected to the data-logger and the thermocouple leads and data-logger are adequately protected from the test apparatus in case of explosion.

18.7.2.4.4 ~~Propane~~Fuel gas pressure and flow is checked and adjusted to the values used during the water calibration described in 18.7.2.3. Video cameras and any other recording equipment are checked and started. Thermocouple functioning is checked and data logging is started, with a time set between thermocouple readings not exceeding 10 seconds, and preferably shorter. The test should not be performed under conditions where the wind speed exceeds 6 m/s, unless additional precautions against side winds are taken. ~~With higher wind speed, additional precautions against side winds are required~~ to avoid dissipation of the heat.

18.7.2.4.5 The fuel gas~~propane~~ burner may be started locally or remotely and all workers immediately retreat to a safe location. Progress of the test is followed by monitoring thermocouple readings and closed circuit television images. The start time of the trial is de.ned by the time at which the flame thermocouple trace T1 first begins to rise.

18.7.2.4.6 The gas reservoir should be large enough to bring the substance to a possible reaction and provide a fire duration lasting beyond total consumption of the test sample. If the vessel does not rupture, the system should be allowed to cool down before carefully dismantling the test set-up.

18.7.2.4.7 The test outcome is determined by whether or not a rupture of the vessel is observed when the test reaches conclusion. Evidence of test conclusion is based on:

(a) The visual and aural observation of vessel rupture accompanied by loss of thermocouple traces;

(b) The visual and aural observation of vigorous venting accompanied by peaking of both vessel thermocouple traces and no substance remains in the vessel; or

(c) The visual observation of decreased levels of fuming following the peaking of both vessel thermocouple traces at temperatures in excess of 300 °C and no substance remains in the vessel.

For the purposes of assessing results, the term "rupture" includes any failure of welds and any fracture of metal in the vessel.

~~18.7.2.4.8 The test is performed two times unless a positive result is observed.~~

18.7.2.5 *Safety Issues*

18.7.2.5.1 Suitable test site selection criteria –

(a) a recommended minimum safety distance of 1km radius from the test area if the test is conducted unprotected;

(b) sufficient distance from public or private areas to eliminate the risk of public exposure to noise, fume and shrapnel;

(c) remote and/or secure enough to prevent unauthorised entry;

(d) minimal significant fire risk in the event of a positive result;

(e) minimal risk from loss of containment issues;

(f) adequate capacity for safe storage of test products; and

(g) appropriate approvals, notifications and awareness.

18.7.2.5.2 Test precautions –

(a) if line of sight to the test is not possible, a remote observation system should be used to observe the test from the recommended safe distance;

(b) re-entry time to the test area should be set at a recommended minimum of 150 minutes if the test is considered still active;

(c) appropriate personal protective equipment (PPE) should be provided. Precautions should be taken to minimise risk of exposure to elements, fire, fuels, hot objects, fumes, etc.;

(d) risk of vehicle damage due to shrapnel on the ground during re-entry to test area.

~~18.7.2.5~~18.7.2.6 *Test criteria and method of assessing results*

The test result is considered "+" and the substance should not be transported in portable tanks as a dangerous substance of Division 5.1 if an explosion is observed ~~in any trial~~. Explosion is evidenced by rupture of the vessel. Once the substance is consumed ~~in both trials~~ and no rupture of the vessel is observed, then the result is considered "-".

~~18.7.2.6~~18.7.2.5 *Examples of results*

Substances	**Result**
76.0 ammonium nitrate / 17.0 water / 5.6 paraffin oil / 1.4 PIBSA emulsifier	–
84.0 ammonium nitrate / 9.0 water / 5.6 paraffin oil / 1.4 PIBSA emulsifier	+
67.7 ammonium nitrate / 12.2 sodium nitrate / 14.1 water / 4.8 paraffin oil / 1.2 PIBSA emulsifier	–
67.4 ammonium nitrate / 15.0 methylamine nitrate / 12.0 water / 5.0 glycol / 0.6 thickener	–
71.4 ammonium nitrate / 14.0 hexamine nitrate / 14.0 water / 0.6 thickener	–